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Institute Notes

OUR fourth fiscal year starts October 1st. The prompt renewal of your membership will encourage your officers and committees in their services for you and our industry.

OUR fourth annual meeting will be held in New York City, Thursday, November 2d, at 10 A.M., place and program to be announced later.

OUR outstanding accomplishments during the current year have been: Membership increase from 140 to 387; inauguration of the industry's first scientific magazine, TEXTILE RESEARCH; *and publication of the book* TEXTILE RESEARCH: A SURVEY OF PROGRESS.

THIS magazine is published primarily as a service to members, but 45 unsolicited outside subscriptions from domestic and foreign scientific organizations, libraries and research men reflect its broadening service and influence.

TO date 941 copies of our book TEXTILE RESEARCH: A SURVEY OF PROGRESS *have been sold. With the considerable foreign sales eliminated this reflects a rather discouraging lack of interest in the subject, for it is a book that should be owned and read by every intelligent and progressive textile man.*

A copy of the booklet TEXTILE RESEARCH PROGRESS, *cataloging all textile research known to be in progress in this country, has been mailed to all members. Extra copies may be had on request.*

WHILE practically every issue of this magazine during the coming year will contain one or more complete semi-annual or annual reports of Textile Foundation researches, the Board of Editors continue to solicit, and will provide space for, reports of individual studies that add to scientific textile knowledge.

A limited number of complete sets of Vol. III, the current number of TEXTILE RESEARCH, *will be available and will be presented to new members of U. S. Institute as long as they last. This is in addition to Vol. IV, starting in November, and the other services for which they qualify automatically.*

TEXTILE RESEARCH

A Critical Study of the Soaking of Silk

Part I.—Prior Work Summarized. Time, Temperature, pH and Ratio of Emulsion Volume to Weight of Silk

By ALFRED N. ROGERS*

BEFORE combining the individual silk fibres into a thread of the desired thickness ("throwing"), it is necessary to soften the layer of sericin or silk glue which coats the fibres. Water alone, besides not being readily absorbed by the fibres, would prove a poor softener and lubricant for the thread. The soaking bath invariably contains an oil, usually olive oil, plus an emulsifier. The latter is obviously essential, since oil and water are immiscible under ordinary conditions. Formerly, sodium soaps of fatty acids served exclusively as emulsifying agents. Recently, however, there has been an increasing tendency to use sulphonated or sulphated oils. Sulphonated oils have the type formula $R-SO_3-Na$, while sulphated oils are characterized by $\begin{matrix} R \\ \diagdown \\ \quad SO_4. \\ \diagup \\ Na \end{matrix}$ R represents a hydrocarbon residue, usually aliphatic, either saturated or unsaturated.

The Theory of Emulsion

It can be understood readily that the character of the emulsifier plays an important role in the type of emulsion formed.

* Mr. Rogers is a Senior Fellow of The Textile Foundation and works under the direction of Dr. E. R. Theis, Lehigh University.

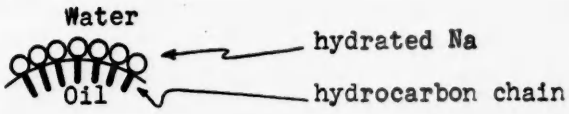
As an introduction to our work a brief presentation of the theory of emulsions will be given. Those interested in a more detailed treatment are referred to "The Theory of Emulsions and Emulsification," by W. Clayton;[1] to the later and more technical publications, "Theory of Emulsions and their Technical Treatment," by Clayton;[2] "Technik der Emulsionen," by O. Lange.[3]

Oil-in-water emulsions can be formed without the aid of a third substance if the concentration of the oil is small. Donnan,[4] Ellis,[5] Powis,[6] and others have shown these dilute emulsions to be readily coagulated by electrolytes. In order to prepare stable emulsions of higher concentrations a stabilizing agent or "emulsifier" must be present. The emulsifier determines not only the stability of the emulsion but also its types, *i.e.*, whether it is an oil-in-water or water-in-oil emulsion. It was formerly thought that the type was determined by the relative proportions of the two phases. In 1910, Walter Ostwald[7] suggested than an oil-in-water emulsion automatically inverts to the water-in-oil type when the oil concentration exceeds 74%, which corresponds to the closest packing of spherical balls. He had overlooked the fact, however, that Pickering[8] had prepared emulsions in which the dispersed phase occupied 99% of the total volume. This would indicate that (*a*), the globules of the dispersed phase can be distorted from their spherical shape; (*b*), the type of emulsion is dependent on something other than concentration. Clowes[9] pointed out that alkaline soaps usually form oil-in-water emulsions while alkaline earth soaps (*e.g.*, of Ba, Mg, or Zn) form emulsions of the water-in-oil variety.

The Heteropolar-Molecule Theory

These and similar observations led to the heteropolar-molecule theory of Langmuir and Harkins, which has been further developed by Hildebrand.[11] According to this theory, an emulsifier must consist of a molecule which is polar (therefore, water soluble) on one end, non-polar (oil soluble) on the other, and long enough to permit one end to be entirely unaffected by the field from the other. Thus in sodium soaps, R-COONa, the -COONa is supposed to jut out into the water, where the hy-

dration of the Na causes a bulging of the interface to form an oil droplet:



Here, obviously, we have an oil-in-water emulsion. The formation of water-in-oil emulsions by soaps of alkaline earth metals is explained:

The portions of the molecules extending from the droplets in either case form a sheath, preventing actual contact of the droplets on collision and thus preventing their coalescing.

This "wedge theory" of Hildebrand has been criticized severely because resins, which have no polar groups, as well as solid powders, are often excellent emulsifying agents. H. R. Kruyt [12] remarks that, at ordinary temperatures, soaps are colloidally dispersed, as are nearly all good emulsifiers. He is in favor of explaining the effect of emulsifiers on the basis of their wettability by oil and by water, respectively. If the emulsifying agent is more readily wetted by water or, as Bancroft puts it,[13] is more soluble in water, it is submerged to a greater extent in the water phase. This causes a bulging of the oil droplet to form an oil-in-water emulsion as shown:

In this explanation the protecting sheath, which prevents coalescing of the droplets, consists of micelles of the emulsifier. If the interface were to curve in the opposite direction, and water be the dispersed phase, collision of the globules would

permit contact of the dispersed phase with consequent coalescing of the droplets. Conversely, a substance more readily wetted by oil (or more soluble in oil) forms a water-in-oil emulsion.

McBain's researches on the electrical conductivity of soap solutions [14] is interpreted by Kruyt (loc. cit.) as pointing to a soap micelle of which the surface molecules are ionized. This is essentially the same as the molecular-emulsifier theory originally introduced, except that the molecule is replaced by an ionic micelle.

The Sulphonated Oils

The above discussion holds in every detail for sulphonated oils as emulsifying agents. If we wish to accept the last explanation offered, we must consider a sulphonated oil micelle as being ionized on the surface:

$$R - SO_3 - Na \rightarrow Na^+ + R - SO_3^-$$

Because of the weakly acid nature of the fatty acids, their sodium soaps dissolve to form alkaline "solutions" of pH approximately 10. Any attempt to lower the pH liberates free fatty acid and makes the soap useless as emulsifier. To employ this alkaline emulsion in treating delicate fabrics may cause permanent injury to the fibre. Sulphonated oils, on the other hand, possess the strongly acid $-SO_3^-$ group. They can be made into emulsions of any desired pH, from a very low value up to 11 or 12, depending on the extent of neutralization of the acid groups (with NaOH). Thus, they can be used in acid or neutral solutions. Although Harris showed [15] that silk finished in alkaline solution is most stable to light, it is known that silk fibre is weakened by alkalies, for alkalies hydrolyze proteins even more readily than do acids. Except for fabrics which are to be exposed constantly to sunlight, therefore, sulphonated oils should be superior to soaps.

As mentioned, the oil in the emulsion is taken up by the silk, which is lubricated by it. In this connection, the sulphonated oil serves merely as emulsifier in order that the oil may be held dispersed in the emulsion in droplets fine enough to be absorbed readily. The sulphonated oil, however, serves another

purpose. Much of the softness and pliability of the silk in manufacture, as well as the strength and elasticity of the finished thread, depends on its moisture content. In addition, the ease with which it is boiled off and the readiness of penetration of the dye liquor are controlled by its wettability. Here the adsorbed or combined sulphonated oil serves as wetting agent. The strongly polar $-SO_3Na$ groups of the sulphonated oil attract and hold water and raw oil micelles on the fibre. Whether we term this attraction of water hydration of the adjutting ions, or adsorption, is a matter of small consequence.

Effect of pH on Oil Absorption

One of the variables investigated in this research was the effect of pH on oil adsorption. Why should one expect the acidity of the emulsion to have any effect? In answering this question, we must first consider that silk is a protein and that proteins are amphoteric, being capable of ionizing either as acid or as base. In acid solutions, it behaves as an alkali; in alkaline solution, as an acid. In the former case, it is positively charged, tending to move toward the cathode; in the latter case it is negatively charged, tending to move toward the anode. The pH at which its ionization as acid just equals that as base is known as its isoelectric point. Here, its electrophoretic velocity is zero. By electrophoretic measurements, Harris [16] determined the isoelectric point of silk as pH 2.5.

The effect of the pH of the medium on the charge of a protein is shown by the curve marked "Charge" (Fig. 1). Below the isoelectric point, the fibre, being positive, should show a strong tendency to absorb negative ions, this tendency decreasing to zero at the isoelectric point (curve IIA). Curve 1A shows adsorption of positively charged particles at pH values greater than the isoelectric point. Instead of considering the ionization of the fibre itself, we may look at the charge as originating from the adsorption of hydrogen or hydroxyl ions. Lachs and Michaelis [17] and Estrup [18] showed that an anion is adsorbed more readily in the presence of a readily adsorbed cation. This principle has found practical application in dyeing. Actually, substances which have a strong attraction for the protein will follow the general trend of the adsorption curves but will not cease to be adsorbed at the isoelectric point.

The effect of pH on the adsorption of dyes has been shown by Briggs and Bull,[30] whose data indicate a decreasing but, nevertheless, appreciable take-up after the isoelectric point is passed. Davison [19] obtained qualitative results confirming these observations.* This condition is shown by curves IB and IIB in the same figure. Since sulphonated oils are strongly adsorbed by silk, it was predicted that the pH-adsorption curve would be similar to IIB. Furthermore, removal of the readily adsorbed H^+ from acid emulsions should raise the pH, while extraction of OH^- by the silk from alkaline emulsions should lower the pH of the latter. How these predictions were fulfilled will be seen in the discussion of our results.

We would have made this same prediction concerning change in pH had we used soaps of fatty acids instead of sulphonated oils, provided the pH range did not extend too far above or below pH 10. Actually, a decrease in pH of soap-stabilized emulsions was observed for silk,[25, 26, 27, and 28] and also in the case of wool.[29] Hart [25] attributes the decrease to a combination of the sodium of the soap with the sericin to form a definite chemical compound. According to the theory outlined above, the soap is partially hydrolyzed into free fatty acids and NaOH. Since the solution is alkaline the fibre tends to adsorb the OH^- ion; and, of course, the sodium accompanies it. We believe that a series of determinations would show that no stoichiometric relation existed. But whether we call this process chemical combination or adsorption is immaterial; for many cases of adsorption may be considered as a reaction involving only surface molecules.

The Hydration of Silk

Very little work has been done on the oil soaking of silk. At Lehigh, some research has been conducted in this field.[21] This work was devoted primarily to measurement of particle size of the emulsion and to hydration of the silk. The results are summarized best in Marshall's own words: [22]

"Silk has been hydrated with various types of emulsions and other solutions. It has been found that hydration is greatest in acid solutions, medium in neutral solutions, and smallest in alkaline solutions.

* Lately, H. A. Neville and C. A. Jeanson [20] obtained additional quantitative results on the dyeing of wool, also agreeing with these data.

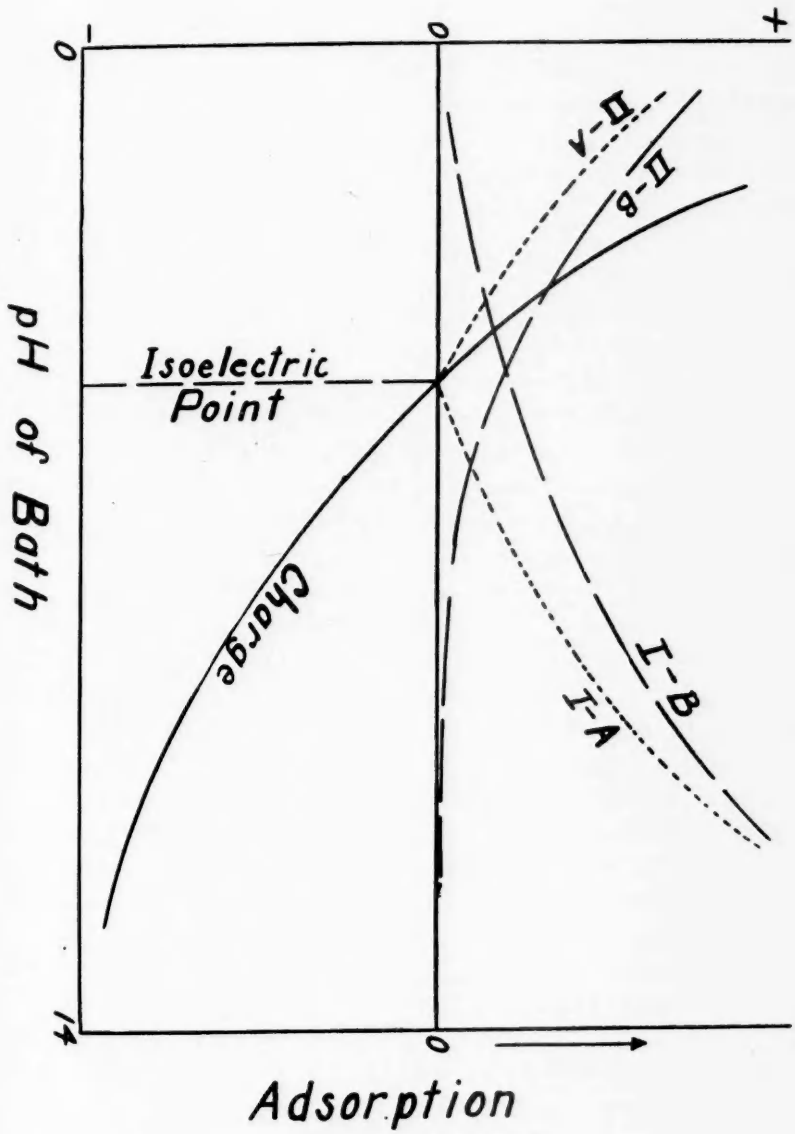


FIG. 1. Charge on Fibre

"The greatest hydrations have been gained using the large 2200 c.c. type dilatometers, where the silk was not as crowded as in the 700 c.c. type dilatometers.

"The most stable soap, oil and water emulsion was found to be one in which .25% of soap was employed as the emulsifying agent.

"Sulphonated oils and combinations of sulphonated oils and other oils have been used to make emulsions; these emulsions were found to be more stable than soap, oil and water emulsions.

"Small concentrations of triethanolamine have been used in sulphonated oil combination to form emulsions. Emulsions made in this manner were found to be very stable and the particle size very small.

"The best temperature for hydrating silk has been found to be 37.8° C. At this temperature sulphonated oil and triethanolamine emulsions were found to reach equilibrium in a period of five hours time. This then cuts the soaking period in half.

"Hydration has been found to increase as temperature decreases; however, the time needed to reach equilibrium becomes greater as the temperature is decreased. When the temperature is lowered there is a lag period introduced.

"As hydration increased the corresponding boil-off values were found to decrease. Therefore, maximum hydration is not a measure of gain in weight where the boil-off method is employed."

Snell and Kimball [23] summarized some of the work done in this field. They say that, although Neville and Marshall show that soap as emulsifier causes greater hydration and gain in weight and acts more quickly, sulphonated oils are preferred because they form more stable emulsions.

It is noted also that increasing alkalinity of the bath improves plasticity up to pH 10. Too much alkali, however, makes the sericin harsh. Also, too much alkali causes "cream separation." On removing the silk some of this scum sticks to it, making it sticky, hard to handle and hard to wet.

Snell and Kimball say oil does not react in any way with the silk but is deposited on it, giving a lubricating effect. Hart and Searell [24] say that the deposition amounts to 19.7% of the neutral fat in the original liquor.

Snell and Kimball think that oil absorption and hydration (dilatometer) give no measure of softening effect. One should test elasticity and pliability of tram, as done by Julius Kayser & Company.

The Important Factors in Silk Soaking

When any protein is treated with an acid or an alkali, chemical combination takes place and may be illustrated:

$$\begin{matrix} -COOH \\ R \\ -NH_2 \end{matrix} + HCl \rightarrow R \begin{matrix} -COOH \\ \\ -NH_2.HCl \end{matrix}$$

or

$$\begin{matrix} -COOH \\ R \\ -NH_2 \end{matrix} + NaOH \rightarrow R \begin{matrix} -COONa \\ \\ -NH_2 \end{matrix} + H_2O$$

The biochemist is familiar with these reactions and no further comment need be made. When, however, a protein substance such as silk is immersed in an oil-water emulsion, the silk is said to absorb the oil. If then the oil-treated silk is carefully dried and extracted with ether, a portion of the absorbed oil is removed. The portion of oil removed by the extraction process is only a small part of the oil taken up by the silk during its immersion in the oil-water emulsion. The portion of oil removed during extraction will be known in this report as the Free Oil. If the silk, after extraction with ether, is then hydrolyzed with 20% hydrochloric acid and the hydrolysate (amino acids, fatty acids, etc.) then extracted with carbon tetrachloride (to remove liberated fatty acids), the oils that were combined with the silk proteins will be removed. The portion of the oils thus removed will be known in this paper as the Combined Oil. The addition of the two extractions is known as the total oil.

In the process of manufacture of silk commodities the raw silk is first soaked either in vats or in patent machines in an oil-water emulsion of one type or another, in order that the silk may be thrown and knitted. This soaking process in silk fabrication is little understood and the technical information concerning it very meager. In making a critical study of the soaking of silk the following factors must be taken into account:

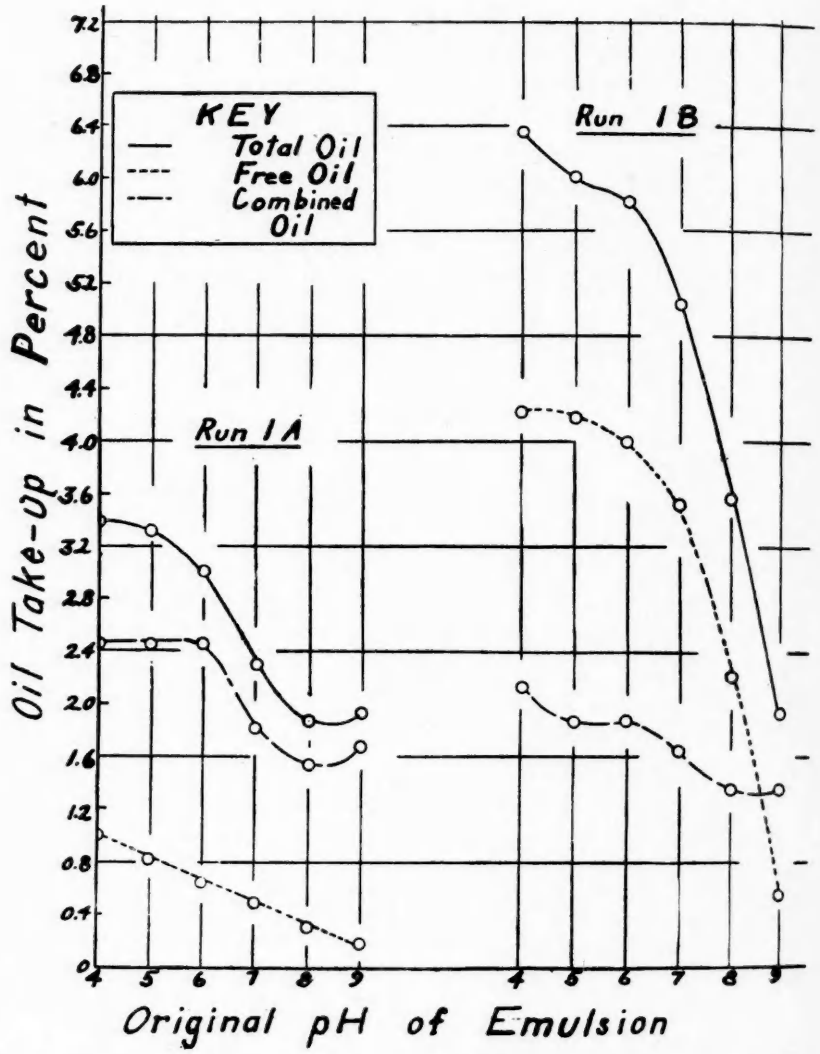


FIG. 2. Showing Effect of Hydrogen Ion Concentration Upon Oil Take-Up

(1) The ratio of volume of oil emulsion to weight of raw silk.
(2) The effect of temperature upon oil take-up.
(3) The effect of hydrogen ion concentration both in the initial and final states.
(4) The effect of various types of oils, with special regard to unsaturated fatty acids, unsaponifiable matter, etc.

(5) The effect of various emulsifying agents upon the resultant oil take-up.
(6) The effect of free and combined oils upon the character of the resultant silk.
(7) The effect of time upon silk soaking.

In this present paper only the effect of time, temperature, hydrogen ion concentration, and ratio of volume of emulsion to weight of silk will be considered. In reports to follow, the other factors will be taken up and discussed in detail.

The Effect of Hydrogen Ion Concentration

When raw silk is soaked in oil-water emulsions of different hydrogen ion concentrations, the amount of free oil and the amount of combined oil taken up varies with the pH value of the emulsion. As a general rule the free oil take-up decreases with an increased pH value of the soaking emulsion. The oil combining with the raw silk decreases also with the increase in pH value, but this decrease is not as marked as is the case with the free oil take-up. Since the total oil is the addition of free and combined oil, the values for the total oil take-up of the silk naturally follow those of the free oil. Fig. 2, Run 1A shows the effect of hydrogen ion concentration of a sulphonated oil-water emulsion upon the free oil, combined, and total oil take-up of raw silk. The combined oil reaches a maximum value at pH 6 and apparently holds this value on the acid side; when the emulsion is made alkaline, the combined oil value decreases. Run 1B shows graphically the results obtained for a sulphonated oil-neutral oil-water emulsion. Comparing 1B with 1A, it is seen that, though neutral oil has been added (as a diluent) to the sulphonated oil, this neutral oil affected the combined oil take-up very slightly, but affected the free oil take-up greatly, especially in the acid range. At a pH value of 9, the free oil, combined oil, and total oil take-up is essentially that of the sulphonated oil alone. Analytical data corresponding to the graphs 1A and 1B are given in Table I.

These results agree with our predictions (as given in the introductory discussion), and with our picture of the mechanism of oil take-up by silk. As has been pointed out, silk, being a

TABLE I

Showing Effect on Oil Take-up of Hydrogen Ion Concentration of Emulsions

(*Sulphonated Oil-Water System*)

pH	% Free Oil	% Combined Oil	% Total Oil
4	0.97	2.45	3.42
5	0.82	2.52	3.34
6	0.64	2.40	3.04
7	0.50	1.83	2.34
8	0.32	1.54	1.86
9	0.23	1.67	1.90

(*Sulphonated Oil-Neutral Oil-Water System*)

pH	% Free Oil	% Combined Oil	% Total Oil
4	4.22	2.14	6.36
5	4.18	1.82	6.00
6	3.97	1.85	5.82
7	3.50	1.63	5.14
8	2.20	1.37	3.57
9	0.57	1.35	1.92

protein, should have a positive charge in liquids more acid, negative in liquids more alkaline, and neutral at the isoelectric point. A negative ion such as $R\begin{cases} COONa \\ SO_3^- \end{cases}$ should be most strongly adsorbed in acid and least in alkaline media. The fact that the combined-oil curves are similar to the pH vs. dye-adsorption curves shown indicates that our oil-combination is closely related to adsorption.

That combined oil does not equal zero in a medium of pH 2.4 (the isoelectric point of silk) indicates that the forces with which we are dealing are stronger than merely adsorption. So great is the attraction for the silk that, even when the fibre is negatively charged, we still have combination. In strongly acid solutions we would suspect that the curves would become irregular, for the acid attacks the silk and alters its chemical composition. In very alkaline solutions, which attack sericin very readily (cf. boil-off procedure), there is a rise in combined oil. It is likely that the loosening of the sericin exposes more of the fibroin to the emulsion (a possible explanation for the effect of bacteria in causing the final rise in the time-absorption curves).

It will be seen also that the free oil take-up decreases sharply with increasing alkalinity of the emulsion. This is an added example of the parallelism between free and combined oil noted below and supports the depicted mechanism of free oil take-up.

It will be seen also that addition of more raw oil to the emulsion raises the free oil take-up, but has practically no effect on the combined oil. This contributes additional support to our postulated mechanism of reaction: free oil would not be expected to have any effect on the adsorption of negative ions on to positive groups of the fibre molecule. The marked increase in adsorption of free oil seems to lead to the conclusion that the latter is mostly raw oil, held to the combined oil sheath by weak chemical bonds or secondary valences.

When raw silk is immersed in oil-water emulsions of varying hydrogen ion concentrations the pH value, during the soaking, changes, shifting on the acid side toward the alkaline side, and upon the alkaline side to that of the acid range. This is what would be expected, since raw silk fibre is an amphoteric protein able to combine either with acids or alkalies. Table II and Fig. 3 show this shift in pH value for an oil-water emulsion.

TABLE II

Showing Change in pH Value of Emulsions during Soaking.

Original pH	Final pH
4.0	5.5
5.0	5.7
6.0	6.0
7.0	6.2
8.0	6.9
9.0	7.5

The Effect of Temperature Upon Soaking

Usually the chemist considers temperature as of great importance in increasing the activity of chemical reactions. It is usually found that an increase in temperature causes an increased reactivity. In most cases the chemist considers an increase in temperature of 10° C. as causing a doubling of the reaction rate. Figure 4 and Table III show that as temperature is increased, the free and combined oil take-up is also in-

creased but not as one would expect from a molecular reaction. The fact that there is only a slight increase, as noted on Figure 4, might be explained by the reason that the kinetic energy of the molecules does not have as much effect as if it were molecular reaction, due to the fact that in this case we have hundreds of molecules dispersed in a drop of oil emulsion; and this, in turn, means that only the surface molecules are available for reaction;

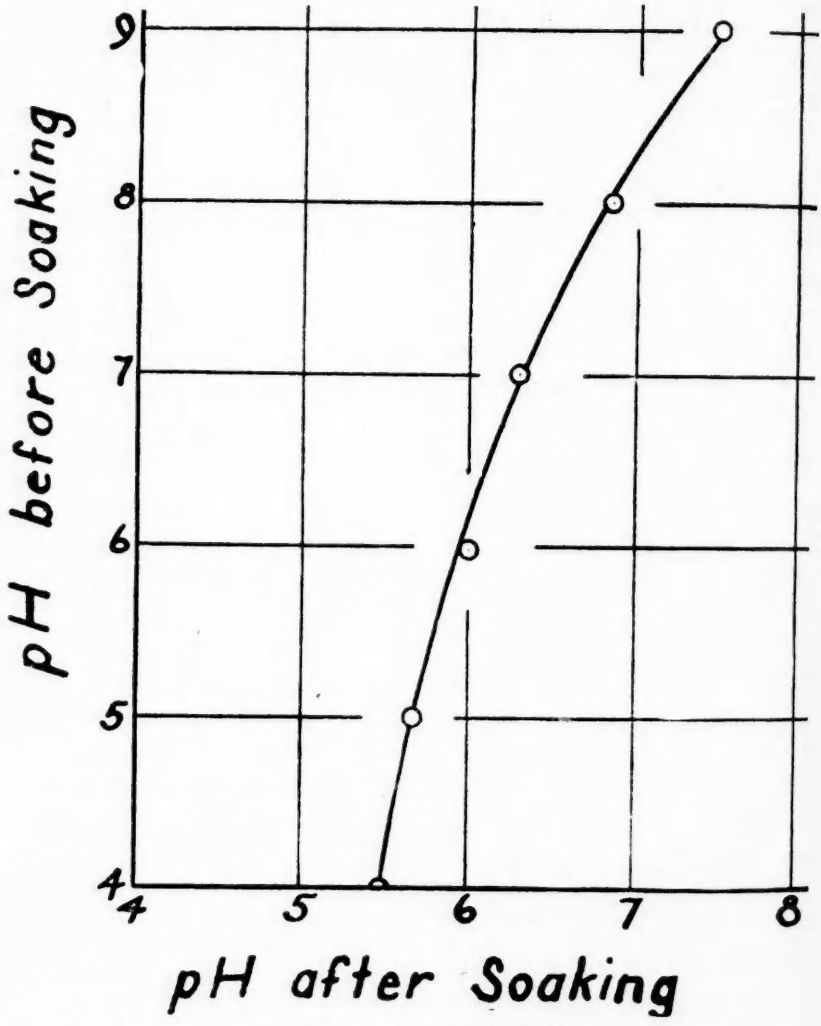


FIG. 3. Showing the Change of pH of Soak Liquors after Soaking

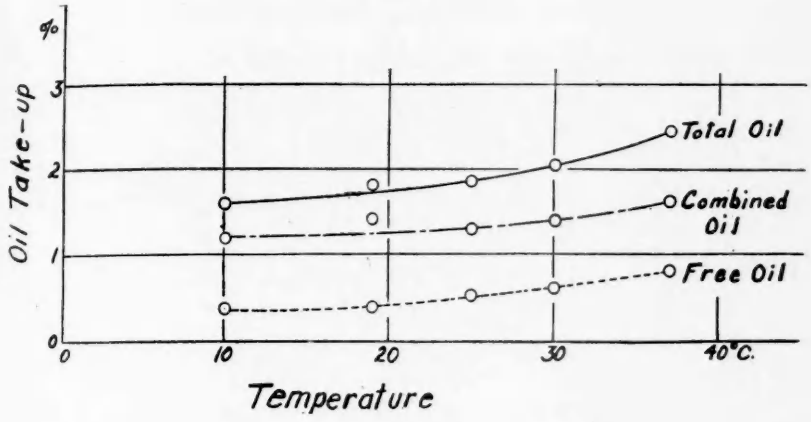


FIG. 4. Absorption-Temperature Curves

TABLE III

Showing Effect of Temperature Upon Oil Take-up

Temperature	*% Free Oil*	*% Combined Oil*	*% Total Oil*
10° C	0.38	1.21	1.59
19	.40	1.42	1.82
25	.54	1.32	1.86
30	.65	1.40	2.05
37	.83	1.64	2.47

Note: A 0.5% emulsion was used. 300 c.c. of this emulsion (equivalent to 1.5 grams of sulphonated oil) used for each 10 gram sample of silk. All emulsions adjusted to pH 7.00 ± .05 before starting.

and thus only a small part of the increase in kinetic energy manifests itself in increased reaction rate. Such a theory might readily explain the total and combined oil take-up but would in no manner explain the free oil increase with temperature. With temperature increase the dispersion of the oil within the water phase would be increased, and thus, due to smaller particle size, increase the penetration of the free oil into the silk fibre itself, thus causing a greater take-up of free oil. In the second place, at the warmer temperature a softening of the sericin might occur and, due to the softening of this gummy material, aid the penetration of the free oil into the silk fibre.

The Effect of Volume Ratio on Soaking

If a definite amount of raw silk is soaked in varying quantities of the emulsion (the emulsion in each case carrying the same weight of oil regardless of volume), it would be expected that the oil take-up by the silk would vary directly with the volume. Figures 5 and 6 and Table IV show the data taken

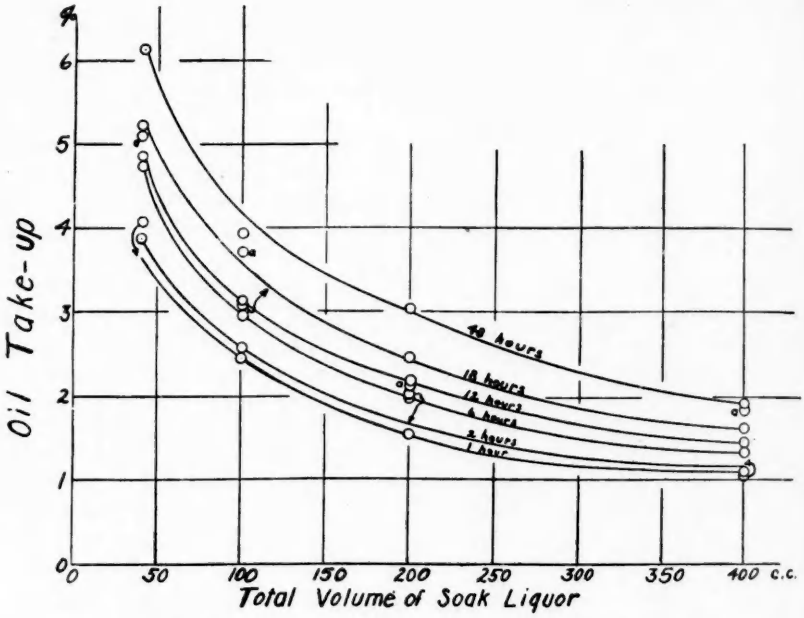


FIG. 5. Absorption-Concentration Curves for Total Oil

TABLE IV

Showing Effect of Time and Concentration on Oil Take-up

Run II

Total volume of emulsion per 10 grams silk—40 c.c.

Time	*% Free Oil*	*% Combined Oil*	*% Total Oil*
1 hour	2.24	1.82	4.06
2	1.87	1.99	3.87
6	1.93	2.80	4.73
12	1.93	2.92	4.85
18	2.37	2.85	5.22
24	2.26	2.83	5.09
48	3.12	3.02	6.14

Run III

Total volume of emulsion per 10 grams silk—100 c.c.

Time	*% Free Oil*	*% Combined Oil*	*% Total Oil*
1 hour	0.68	1.77	2.45
2	0.78	1.77	2.56
6	0.86	2.09	2.95
12	1.14	1.94	3.09
18	1.07	2.06	3.14
24	1.46	2.24	3.70
48	1.74	2.20	3.94

Run IV

Total volume of emulsion per 10 grams silk—200 c.c.

Time	*% Free Oil*	*% Combined Oil*	*% Total Oil*
1 hour	0.41	1.13	1.55
2	0.66	1.36	2.02
6	0.60	1.40	2.00
12	0.80	1.38	2.18
18	0.71	1.74	2.45
24	0.72	1.42	2.14
48	1.26	1.76	3.03

Run V

Total volume of emulsion per 10 grams silk—400 c.c.

Time	*% Free Oil*	*% Combined Oil*	*% Total Oil*
1 hour	0.22	0.88	1.11
2	0.23	0.82	1.05
6	0.32	1.02	1.34
12	0.34	1.10	1.45
18	0.45	1.16	1.62
24	0.46	1.32	1.78
48	0.58	1.33	1.91

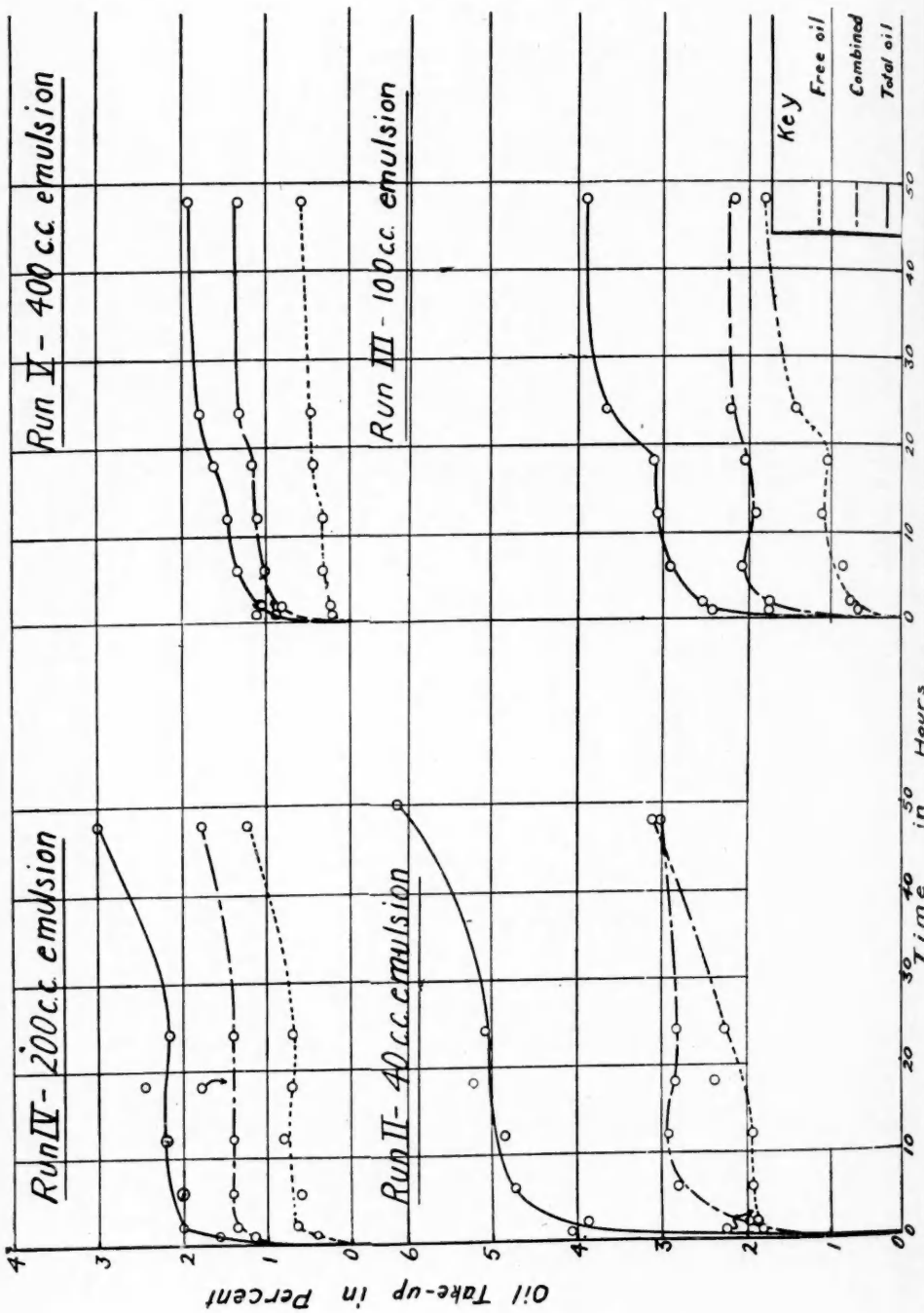
Note: In all the runs, the ratio of oil to silk was the same: 1.5 grams dry oil to 10 grams silk.

The original pH of all the emulsions was adjusted to $7.00 \pm .05$.

As was expected from our previous determinations, the pH of all the soak liquors dropped to a final value of 6.5 to 6.7.

Same oil used as in determination of data for Table III.

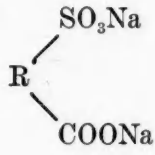
from actual experiments. The following conclusions can be deduced from the figures:


Run V - 400 c.c. emulsion
Run III - 100 c.c. emulsion
Run IV - 200 c.c. emulsion
Run II - 40 c.c. emulsion
Key
Free oil
Combined
Total oil
Oil Take-up in Percent
Time in Hours

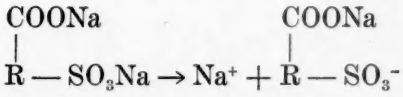
1. An increase in volume causes a lessened oil take-up for both free oil and combined oil and, consequently, total oil. Thus, it is seen that an increased concentration of oil causes a more rapid and increased amount of oil to be taken up, which is in line with the mass action law.

2. The curves in all cases show higher values for combined oil than free oil. It is readily seen that the ratio of combined oil to free oil changes with the volume of emulsion used and thus changes with the oil concentration in the emulsion. In the case of Run II, Fig. 6, this ratio is 1.25 to 1; for Run III, the ratio is 1.5 to 1; for Run IV, the ratio is 2.0 to 1; and for Run V, the ratio is 2.8 to 1.

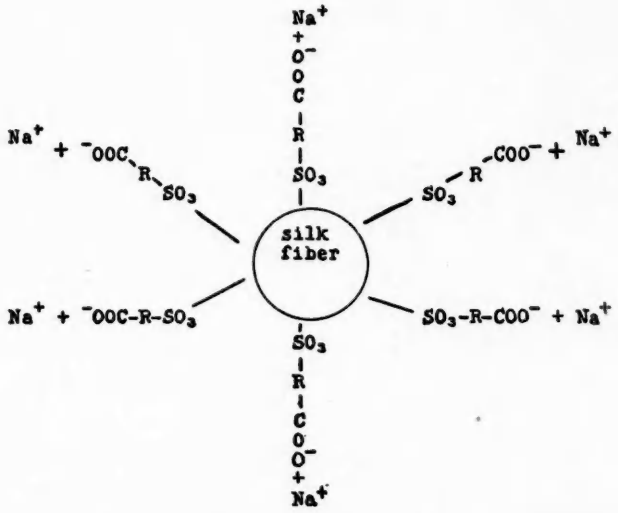
3. By a careful study of the curves in Fig. 6, it is seen that if the percentage of free oil is subtracted from that of the combined oil, the difference in approximately all cases is 0.75 per cent, which would seem to indicate that the amount of free oil taken up by the silk is directly dependent upon the combined oil taken up by the silk. This free oil dependency would further indicate a possible explanation of the relation between the oil take-up and the silk fibre. We can represent the molecule of neutralized sulphonated oil as



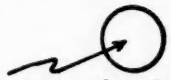
where R is a hydrocarbon residue. Since the sulphonated end of the molecule should be strongly polar in character, we can picture the principal ionization as



Now, because of its negative electrical charge, the resulting ion is absorbed upon the silk, being attached through the $-SO_3^-$ group. The oil thus adsorbed is our "combined oil." The adsorbed oil can now be further ionized at the carboxyl linkage, leaving some such residue as pictured:



The outer sheath of Na^+ ions might be thought of as presenting a weak secondary positive valence toward the solution. Any negative ions wandering near the fibre would be attracted to it and weakly held. Thus, if an additional sulphonated oil ion were to be attracted or a neutral oil ion, attracted through residual secondary valence bonds, we would have silk fibre


$$—SO_3—R—COO^- \text{———} Na^+ - - - SO_3—R—COONa$$

where the dotted bond indicates a weak chemical attraction. Since the attraction for the additional molecule is small (due either to a shared valency bond or secondary valency bonds), the weakly held molecule would be easily removed by ordinary oil solvents, while the strongly held molecule attached directly to the fibre would not.

4. Fig. 6 shows that, in most cases, the curves approach a maximum value and seem to attain an apparently constant value. This constancy, however, obtains only for a short interval, at the conclusion of which the curves show a sudden upward trend, which would indicate reactions other than those of a purely chemical nature. This upward trend might be explained from a bacterial and enzymatic viewpoint. The portion of the curves up through the apparent constant value might be considered as representing a bacterial lag period, in which no bacterial activity

is evidenced. At the point in the curve where the upward trend begins, the start of the period of bacterial activity occurs. That such a theory holds is evidenced by a distinct odor from such a time on. When bacterial action begins the silk fibre is attacked, which action in turn affects the oil take-up of the silk fibre.

Summary

In this report we have studied in detail the effect of time period of soaking; the effect of volume ratio of emulsion to weight of silk; the effect of temperature; the effect of hydrogen ion concentration upon the oil take-up of the silk fibre. We have postulated new theories to account for the oil which is combined or adsorbed by the silk fibre. In this work there is shown to be a definite relation of the combined oil to the free oil adsorbed.

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Legal Status of Inventions of Employed Researcher *

A New York concern employed a chemist to devise certain methods of treating cloth materials. His service in that capacity was satisfactory, as far as the records show. Later he obtained letters patent on a method of developing a distinctly new and different material.

The concern which employed him originally brought suit to compel him to assign to it the patent covering his invention, on the ground that he was employed by it to find the very thing he invented and paid for that very service.

Justice Meier Steinbrink held that he could find nothing in the evidence to show that the defendant was "employed specifically for the thing that was thereafter accomplished" and said:

> Let me submit this suppositious case: I go to a chemist with the idea, which I transmit to him, of dispelling heavy fog at sea by the release of a heavy chemical spray of gas. I employ him for this. In the course of his experimentation the chemist finds a way of chemically treating the glass of marine binoculars so that through them vision in fog is possible. Will it be argued that in private industry employment for the former purpose entitles me to an assignment of the patent on the latter method simply because it was discovered in the course of experimentation in the former search?

Justice Steinbrink dismissed the complaint and brought joy to those research specialists and inventors who are tied to contract to give employing concerns full rights to whatever they may discover. Because the concern had paid the chemist for his services, the court absolved it from costs of the action.

The decision, hailed in legal circles as a good combination of equity and common sense, is the law of the State unless and until the higher courts reverse it. It means tighter drawing of agreements between employer and employee if the latter is to agree actually to sell all he may discover in research for something else for which his employers may have immediate use.

* From the *Syracuse Post-Standard.*

Protective Effects in Textile Deterioration and Dye Fastness

Part I.—Conclusions of Literature Survey; The Oxidation of Mannitol

By DONOVAN J. SALLEY*

IN the original application for a Fellowship, it was proposed to study the protective effect of inhibitors on (*a*), the deterioration of textiles by atmospheric action, and (*b*), the prevention in dyed materials of the color changes accompanying such atmospheric action. The deterioration by the atmosphere was attributed to an oxidation reaction, and it was supposed that color changes were brought about by the accompanying oxidation, degradation, and decomposition reactions of the dye. It was pointed out that the oxidation of numerous and widely varied substances such as aldehydes, fats, alkaloids, rubber, etc., could be arrested or accelerated by the addition of traces of various substances, and certain empirical facts in the literature indicated that the same phenomena also were operative in textile deterioration.

Thus the broad subject proposed for research was in itself part of the general field dealing with autoxidation, chain reactions, and negative and positive catalysis. In the research it was desired not so much to compile a list of deterioration preventatives as to make a thorough study of the oxidation process and the mechanism of the inhibitory action. Thus an investigation of the oxidation of textiles and related substances not only might add information of value to the textile industry, but also might contribute to the general theories of oxidation mechanism.

Main Aspects of Subject

With such a viewpoint in mind, the first period of the fellowship tenure was devoted to a search of the literature. The

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various aspects of the subject could be grouped under several main heads:

1. The action of oxygen on cellulose, alkali-cellulose, and undyed fabrics.
2. The action of light on cellulose and undyed fabrics.
3. The action of light on dyes and dyed fabrics.
4. The protection of dyes and dyed fabrics.
5. The oxidation of substances related to cellulose, and the general theories of mechanism of autoxidation and inhibitory action.

This review of the literature showed that a considerable number of studies had been carried out on the above mentioned subjects, and that a number of facts of great interest and importance had been unearthed as a consequence. The general criticism of the great majority of these researches is that they yielded results entirely qualitative and empirical in nature. Usually also, the control of the variables was not rigid, and no guiding principle was followed in the conduct of the investigation. Interpretation of the results was consequently made very difficult.

Conclusions of Literature Study

However, certain general conclusions could be reached from a study of the combined information.

1. Alkali cellulose absorbs oxygen from the air slowly at ordinary temperatures, and the oxidation is much accelerated by the addition of small amounts of a heavy metal such as iron or copper. This action was known to be important in the xanthate process of rayon manufacture. The mechanism of the oxidation process, or the cause of the catalytic effect of the metal, has not been studied.

2. The deterioration of fabrics and of cellulose is primarily due to photochemical oxidation, as some ten or more investigations carried out during the past fifteen years have demonstrated. Outside of the fact that oxygen is necessary in the reaction, the only other known facts are that light of wave-length shorter than 4000 angstrom units is necessary to effect the change, and that impregnation of the fibre with iron or other

heavy metal considerably accelerates the deterioration. Thus, no information on the actual kinetics of the oxidation process has been sought.

3. In the matter of the bleaching of dyes, several well conducted researches have yielded information of great value. The bleaching has been found to result from an oxidation process, and in two cases the number of molecules of dye reacting per quantum of light supplied to the system has been determined. These quantum efficiencies have been low, and therefore one could expect that prevention of the bleaching of the particular dye could not be effected by small amounts of inhibitor, but that the so-called "internal light filters" would give the best protective action. Other experiments have shown that with certain dyes, decolorization can occur by means of a photochemical decomposition process, no oxygen being required. There is also some evidence that dyes can act as photosensitizers, thereby accelerating the deterioration of their fibre substrate. In still other cases, the dye has been shown to be an inhibitor of the oxidation of the fabric. It is thus evident that there can be a mutual reciprocal action between fibre and dye, but no prediction can be made concerning the possible direction of the action. Protection of dyes and dyed fabrics has been accomplished in a number of instances by the use of small traces of inhibitors added to the dye or impregnated in the cloth, and in other cases by the coating of the dyed fabric with a layer of a substance opaque to light. The former method of inhibition is the more important of the two, and would seem to indicate that the oxidation processes in those cases were chain processes.

4. The oxidation of substances related to cellulose, such as sugars, aldehydes, polyalcohols, etc., has been investigated in some detail in various instances, and considerable aid is gained by a study of such results. Concerning the general theories of oxidation mechanism, in some cases the course of the process has been very definitely established. Thus the oxidation of sodium sulfite and benzaldehyde proceeds through a chain of reactions, and the nature of the chain has been recently the subject of much investigation by Haber and his co-workers. In other cases it seems evident that reaction proceeds through building up appreciable concentrations of peroxides, while in

still other examples, the oxidation is brought about by an oxidation-reduction couple set up through the agency of an added metal catalyst.

Apparatus For Mannitol Oxidation

In seeking a possible starting point for an experimental investigation, our attention was drawn by the similarity of the oxidation of polyalcohols in the presence of iron and alkali, and the oxidation of alkali cellulose. The first experimental work in attempting to establish a reaction mechanism has therefore been carried out on the oxidation of mannitol.

Two kinds of apparatus, one of the shaker and the other of the bubbler type have been used in this work. The shaking machine was previously used by Alyea and Backstrom, and consists of an inclined turntable operated by a motor-driven eccentric. The reaction vessel is clamped to the shaker and connected by a short length of rubber tubing to a gas burette from which the volume of oxygen, absorbed at constant pressure, can be read. The shaker with the reaction vessel is mounted in a thermostat adjustable to any desired temperature. To facilitate photochemical measurements, a window was cut in the side of the thermostat directly opposite the reaction vessel, and covered with a quartz plate. This system has the advantage that it is rugged and is easily operated. It cannot be used at pressures other than atmospheric, nor is it suitable for use if gaseous oxidation products appear during the reaction. Furthermore, although it can be used for general photochemical work, quantum yield measurements are not easily carried out in it.

The bubbler type apparatus which has been developed consists of a circulating pump, a gas burette, and the bubbler proper in which reaction takes place. The bubbler consists of a porous alundum plate mounted in a removable glass reaction vessel which contains the reacting liquid. The circulating pump forces oxygen through the alundum plate, which breaks up the gas into very fine bubbles and so insures complete saturation of the liquid with the gas. A gas burette connected to the system permits the determination of the volume of oxygen absorbed during the reaction. This apparatus is well adapted

for photochemical work and for the determination of quantum yields. Moreover, any gaseous oxidation products can be removed by suitable absorbents placed in the circulating gas stream. The chief difficulty with the apparatus is that the pressure adjustment in the system cannot be made very accurately because of the pressure fluctuation arising from the circulation of the gas; however, by means of a small auxiliary manometer, this difficulty has been largely obviated.

Results of Mannitol Oxidation Studies

A considerable number of experiments on the oxidation of mannitol have been carried out in both of these types of apparatus, the majority of the runs in the bubbler system. The rate of oxygen absorption by mannitol solutions under similar conditions of reaction is practically the same for both types of apparatus, showing that no peculiarities of design or construction are affecting the results. To date, the variation of the rate of reaction with the change in mannitol concentration for thermal reaction, and the variation of the rate with change in iron concentration for both the thermal and the photochemical reaction has been determined. For concentrations of mannitol less than 0.1 millomol per cc., the rate is directly proportional to the concentration; for iron concentrations up to 0.05 millomol per cc., the rate varies in a more complicated manner. The empirical equation which approximately expresses the observed rate of absorption of oxygen is: $\frac{dx}{dt} = \frac{k\,(M)\,(F)}{1 + b(F)}$ where k and b are constants and (M) and (F) are concentrations in millomoles per cc. of mannitol and iron respectively. This shows that the reaction rate is not directly dependent on the concentration of the mannitol-iron complexes which form in alkaline solution, as was stated by Traube in his original work on this oxidation reaction, but rather indicates that the iron becomes increasingly less effective as an accelerator for the oxidation. In the photochemical reaction, the effect of the iron is approximately the same as in the thermal reaction. In our particular set-up, using light from a mercury arc of wave length longer than 3100A°, the photochemical reaction is approximately five to six times faster than the dark reaction.

At the present time, it is not possible to state whether or not the oxidation of mannitol in the presence of iron and alkali is a chain reaction, though some evidence seems to point to that possibility. The existence of a long induction period for the dark reaction, this period being considerably shortened in the photochemical reaction, and completely eliminated by the addition of a small amount of hydrogen peroxide, indicates the presence of a small amount of inhibitors which must be oxidized before the reaction of the mannitol itself can proceed.

This work is being continued, and it is hoped that contemplated experiments on the quantum yield of the reaction, the effect of inhibitors, and the temperature coefficient will yield the information necessary to the interpretation of the mechanism of the oxidation.

Textile School Survey

THE directors of the Textile Foundation have authorized a survey into the personnel needs and the educational facilities for the textile industry. A committee, consisting of Drs. Karl T. Compton, president of Massachusetts Institute of Technology, E. C. Brooks, president of North Carolina State College, and R. E. Doherty, Dean of the School of Engineering, Yale University, was appointed to inquire into the possibilities of such a survey, and based on their recommendations the work is proceeding under the direction of Frederick M. Feiker.

Mr. Feiker has been Director of the Bureau of Foreign and Domestic Commerce for two years. He brings to his new task not only the background of his public service in that post, with its wide contacts in many industry needs and trends, but a highly diversified experience of twenty-five years as an engineer-publicist, editor, association executive, university lecturer and Government official. From his own experience on the Board of Trustees of his Alma Mater, Worcester Polytechnic Institute, and as a member of many educational committees, as well as editorial director of many technical and business publications for the A. W. Shaw and McGraw-Hill publishing companies, including association with the journals *Electrical World, Textile World, Power, American Machinist, Factory, Chemical and Metallurgical Engineering,* etc.

To initiate this undertaking, a conference of the heads of the textile schools was held in June, under the chairmanship of Dr. Brooks. Those present were: J. H. Hanford and Wm. Smith, New Bedford Textile School; E. W. Camp, Alabama Polytechnic Institute; J. B. Bagley, Texas Textile School; C. D. Brandt, Texas Technological College; Thomas Nelson, North Carolina State College; H. H. Willis, Clemson Textile School; C. A. Jones, A. French Textile School; Henry W. Nichols, Bradford Durfee Textile School; Chas. H. Eames, Lowell Textile Institute; E. W. France, Philadelphit Textile School; F. M. Feiker and Edward T. Pickard, secretary of The Textile Foundation.

Progress of Textile Research in Great Britain

Encouraging progress and a few notable achievements are noted in the reports for the year 1931–32 of the British research associations for the textile industry to the Department of Scientific and Industrial Research, from which they receive grants for the partial financing of their work. The reports of the cotton, wool, silk and linen research associations are reprinted in large part, as is a report of fabric research conducted by other British laboratories.

Cotton Industry Research

"SOME eighteen months ago the Association commenced a very fundamental attack on the general problem of opening and cleaning cotton. At first, as so often happens, progress was slow and much time was spent in overcoming unforeseen difficulties; recently, however, great progress has been made and it is not too much to say that research results now being obtained may soon lead to revolutionary ideas which will greatly increase the efficiency of opening and cleaning machinery.

"The spinning plant in the new machinery building has continued to be of great service in the investigation of new cottons, new machinery, and variations in methods of processing. That this work is highly appreciated by the industry is shown by the frequent and largely increased number of requests from spinners and doublers for assistance in practical problems, particularly in the elimination of imperfections in yarns.

Machinery Testing and Study

"The testing of new machines which may prove of value to the industry is an important part of the work of the Shirley Institute, and is not confined to those machines developed by the Association. At present, comparative tests are being carried out on the recently invented inclined-spindle ring frame, and on the application of high-draft systems to speed frames as well as to the ring frame.

"A searching investigation into the mechanism of the Lancashire loom with the object of improving its efficiency has been in progress for some time. This investigation has revealed a number of unsatisfactory features in the existing mechanism and in weaving technique, the remedying of which is being actively pursued. In fact, in some instances, effective remedies have already been found and are being applied in mill practice. Many of even the highest quality cloths produced in Lancashire today contain defects, and some of these can be attributed to the imperfect functioning of the loom. This is one of the many ways in which the work of the Association is tending to make for the production of more perfect products.

Chemical Problems

"The fundamental study of dyestuff solutions, and of the dyeing process is still in a very preliminary stage, although it has been the subject of continuous work during the past year. The necessary antecedent of any such physico-chemical study is the provision of pure dyestuffs and of reliable methods of analysis. This requirement is proving particularly difficult to satisfy, and the whole question of the analysis of dyestuffs may require handling as a major problem. Of the various lines of work mapped out in the last report, the most progress as regards the elaboration of suitable technique has been made in the investigation of the penetration of dyestuffs into cellulose material. A systematic investigation of the absorption of vapours by cotton and rayon is also making good progress, and the results may eventually be found to have theoretical bearing on the problems of dyeing.

"On the more immediately practical side, the effects of the bleaching process on the dyeing qualities of cotton cloth, and on the stability of the so-called fast dyes, have been subjects of further investigation along the lines indicated previously.

"An examination of various factors in the process of mercerisation that influence the dyeing qualities of the mercerised material has proved of great practical value to the hosiery industry and to the suppliers of cotton yarns for that trade.

Work of Testing Department

"The work of the Testing Department may be divided into two parts. There are first of all the 'routine' day to day problems, concerned largely with details of structure of yarns and fabrics and with tests for their breakdown. Analyses of fibres, yarns and fabrics and their strength characteristics have now been systematically developed and this service is greatly appreciated by members. Work in connection with such problems as these may be regarded as routine not because it is of the type which can be carried out at any textile Testing House, not because the increasing familiarity of the staff with many of the problems of the industry is enabling them more and more to diagnose quickly and surely the cause of defects which only a few years ago needed careful and prolonged investigation. It is possible, therefore, in a majority of the inquiries to direct attention at once to the most likely cause of trouble and with the help of the Liaison Staff quickly to advise the firms what steps to take to improve their material or effect economies. As soon as the number of inquiries received in regard to some particular difficulty makes it apparent that interest in that problem is not limited to a few members only, it is the practice of the Institute to publish one or more articles in its Confidential Bulletin, describing as far as possible the cause and cure of the trouble. Side by side with these recurring difficulties, new ones are constantly being received by the Institute; such problems keep the Association in close touch with the Industry and what is perhaps more important still, furnish many ideas for fresh fundamental research. Altogether between one and two thousand individual and special problems are now being submitted annually by members.

"The other part of the Testing Department may be called the development section, where new tests are being devised, and where the possibility

of adapting more refined physical methods to the service of testing is constantly being studied. Many such new tests have been developed; for example, such cloth characteristics as air permeability, water-proofing, wettability, shrinkage, deterioration of strength, lustre, colour, and electrical properties are all being investigated by physical methods. An interesting example of the development of these new testing methods is a machine for determining cloth strength; cloth can now be tested under conditions equivalent to those in a complete structure or continuous sheet of fabric instead of under the present standard artificial condition of a strip with unsupported edges. Preliminary tests on this instrument have, indeed, shown that the standard cloth testing machines now in use in the industry give values for cloth strength considerably less than those given by the new machine under testing conditions much more closely simulating those existing in practice.

"The winning of the cotton industry *as a whole* to a full appreciation of the value to it of scientific research is perhaps the biggest problem facing the Association. An outstanding fact of a year of great activity is that in the trade the prestige of the Association is now very distinctly greater even than it was 12 months ago, and there is every sign that it will continue steadily to increase. There are many hundred firms in all sections of the industry who, as a matter of course, seek the Association's assistance on the more complex problems which arise. Of these firms quite a large proportion now regard close co-operation with the Institute as essential to their own efficiency and technical development. There are several hundred other firms who, in varying degrees, show some interest in the research work and its applications. If the present activities of the Association can be continued and intensified, without any curtailments, for another five years or so, there is little doubt that there will have grown up in the trade such a weight of opinion in favour of research and scientific methods that these will by that time be assured of their rightful place in the future of the Industry."

Wool Industry Research

"To the general public the colour or shade of a fabric is that which possibly has the greatest aesthetic appeal and the fastness of such colours to the various influences which might fade them is of paramount importance. Three common causes of fading are light and atmosphere, laundering, and perspiration.

"During the last year, under the joint scheme with the Society of Dyers and Colourists, work on the standardisation of tests for the fastness of coloured and dyed fabrics has reached its final stages, and for fastness to light and atmosphere, all the 14 standards necessary have now been selected. Standard tests for fastness of colours to laundering operations are being exhaustively tried out prior to publication to the trade. Tests are also being evolved for shrinkage, so that the purchaser will have reliable methods by which to test the performance of materials in these respects.

"In woollen carding, the new type machine has been adopted in certain mills in Yorkshire and comparative tests against ordinary mill practice have proved satisfactory for many types of blends. Further trials are proceeding.

"The production of felts for various purposes, *e.g.*, printers' blankets

for covering rotary presses, has been successfully investigated and as a result of the Association's work, improvements have been made in English felts to enable the manufacturers to meet the demands of the newspaper trade, and so to withstand foreign competition.

"A modification of the loom has led to a practical suggestion to prevent the breaking down of the warp during weaving.

"Practical measures have been suggested for the protection of fabrics against the ravages of moths and it has been clearly demonstrated that many fumigating materials do not afford complete protection, but that such can be secured by means of paper or cellulose covers of suitable design in which fabrics can be wrapped and completely protected for all time.

"The control of atmospheric conditions in standard testing rooms and factories has now been successfully accomplished and already the Research Association's system of control of temperature and humidity has been installed and adopted in a number of mills. Not only this, but the system has been extended and actually utilised in confectionery factories, electrical power sub-stations, as also in testing rooms for factories throughout the world.

"On the worsted side further developments have ensued with regard to the spinning of short fibre materials, resulting in an increased spindle speed."

Silk Industry Research

"A LACK of uniformity in some recent deliveries of raw silk of definite descriptions from one of the producing countries is indicated by the occurrence in the finished goods made from it of defects, such as differences of shade, similar to those that might be caused by the use of silk of different origins; a study in the Association's laboratory of the mean diameters of the constituent filaments of a sample of this defective silk suggested that a gradual change had occurred during the reeling in the character of the cocoons in the basin so that whilst neighbouring portions of the thread might not differ greatly, noticeable differences, for example, in dyeing properties, might occur in widely separated portions. The question has been taken up by the Silk Association of Great Britain and Ireland with those concerned in the producing country. The mean diameters of the filaments of mulberry silk from different regions may differ widely, but it has been observed by the Silk Research Association, in the course of a detailed study of the mechanical properties of silk filaments, that the ratio between the mean diameter and the mean cross sectional area of silk filaments is practically the same in silk from these different regions; the wide differences between the mean breaking loads per filament are due, in part at least, to the differences in the diameter, for the mean breaking loads per mean cross sectional area of samples of silk of different origins are less widely different. The hygroscopic properties of samples of degummed mulberry silk from different places are closely similar. The microscopical study of silk has been facilitated by the introduction by the Association of some new stains to distinguish between fibroin and sericin.

"Various pH values have been given by different observers as defining the hydrogen-ion concentration of solutions in equilibrium with silk fibroin at its isoelectric point. It has now been shown by the Association that the swelling of silk filaments passes through a series of minimum values in solutions of acid reaction over the range of pH values 1–5; the

lowest minimum value occurs at pH 3.8, the value earlier given on chemical grounds by the Association as the isoelectric point, and the pH values assigned by other observers to the isoelectric point are values at which other minima occur. These observations on the swelling of silk filaments have been rendered possible by the development of a technique which allows the change in the swelling to be followed with precision by measurements of the width of the filaments notwithstanding the irregularities in the width. The swelling of silk gut in these feebly acid solutions is similar to that of the natural filaments. The apparent amounts of acid or alkali fixed by silk from solutions of these substances appear to be related to the swelling. These results have practical bearings as well as a theoretical interest.

"Researches in other fields, whilst they have proceeded steadily, do not meantime call for special reference. The interest taken by the members in the work of the laboratory increases—many requests for information have been satisfied, and the staff have assisted several members at their mills in the application of laboratory results on a large scale."

Linen Industry Research

"FLAX factory research is now concentrated on means for overcoming the decline in flax growing resulting from a depressed market coupled with intense foreign competition in the supply of cheap fibre. The greatest hindrance to the setting up of a prosperous flax industry in the British Isles arises from the cost of the excessive amount of hand labour at present necessitated. The general plan now evolved aims at a complete mechanisation of the industry. Detailed schemes are being prepared and the necessary new machinery planned and constructed as quickly as possible.

Yarn Strength and Levelness

"The installation of a fairly comprehensive range of machinery for experimental work on spinning and weaving has greatly facilitated the work of the Institute on the mechanical side. Investigations on the effect of reach in wet spinning have shown that the strength of the yarn from given fibres is directly related to the levelness of the yarn and the fineness of the strands in the yarn. Levelness depends, in addition to level preparing, on the use of the right reach to suit the rove weight, twist, and fibre composition. Irregularity in diameter makes it impossible to produce even, closely woven fabric, of considerable importance in some connections. Also the appearance of some fabrics is considerably affected by yarn regularity. The measurement of diameters has been found to offer advantages over other methods of finding the degree of regularity in that it does not necessitate cutting or destroying the yarn. An entirely new method has originated at the Institute which appears to be free from the objections brought against the other methods of measuring yarn regularity, and so research is now being concentrated upon it.

Weaving and Sizing

"Considerable information has been collected regarding the position of warp breakages during weaving and an analysis of these results shows that there are well-defined regions in the plan section of the loom where breakages are exceptionally numerous.

"Experimental warp sizing trials have been mainly concerned with the standard dressing machines as used in the Linen Trade, namely, the flat Atherton and the cylinder machines. So far attention has been given chiefly to types and concentrations of dressing, and three types of size mixtures have been isolated as being outstandingly good. The exact effect of the presence of a softening agent, such as tallow, applied in various ways and concentrations, has also been successfully studied.

Chemical Studies

"It has been shown that the fastness of some vat dyestuffs on linen is not materially affected by the non-cellulosic impurities or by the presence of oxycellulose. An investigation of the behaviour of cellulose dyed with a range of vat and insoluble azo dyes in chemicking has shown that the majority of orange and yellow and some red vat dyes produce excessive tendering in presence of light, while in the dark the dyed cellulose gives results similar to those obtained with the undyed. A number of insoluble azo dyes exert a protective action during bleaching.

"The solubility number method of estimating the degree of chemical degradation of a cellulose material has been compared with the cuprammonium viscosity method and the results obtained indicate that it may be possible to distinguish between acid and oxidation tendering by using the two methods in conjunction.

"Considerable progress has been made in the chemical investigation of the cutin of the epidermis of the flax plant. This substance is of great importance in bleaching problems as it is the non-cellulosic constituent which is most difficult to remove. A mixture of four fatty oxy-acids has been isolated from the cutin by alkaline hydrolysis.

More Rapid Beetling Possible

"A laboratory beetling machine has been constructed and has been in use for some time in an experimental investigation of the beetling process. It is hoped that it will be possible to obtain valuable information with this machine much more quickly than by conducting experiments on a full-scale machine. An experimental machine designed to obtain in an entirely new way the 'closing up' effect such as is produced by beetling has given promising results, indicating that it may be possible, provided the principle can be embodied in a practical machine, to obtain the desired effect much more rapidly."

Additional Fabrics Research

"THE research workers concerned in the investigation into the destruction of fabrics by micro-organisms, having completed their review of the results obtained from the exposure of fabrics at various stations, have now commenced on a physiological study of the destructive types of micro-organisms found on the fabrics. The research at the Royal Aircraft Establishment on the development of a 'permanent' method of fireproofing fabrics has recently been completed, while the work at the National Physical Laboratory on the action of sunlight on cotton was brought to a conclusion in March last. A report describing the results of these researches is now in course of preparation with a view to publication."

Textile Foundation Researches

Additional subjects that are being studied by research fellows of the Textile Foundation are described non-technically in the reports that follow. Textile Foundation researches similarly described and published previously are as follows: "Effect of Bacteria and Vegetable Oils on Silk"; "Application of Polarized Light to Textile Research"; "Protective Effects in Textile Deterioration and Dye Fastness"; "Spectrophotometric Analysis of Dyed Materials"; "Mildew Prevention and Treatment"; "Orienting Influences of the Benzene Ring"; "Moth-repellent Dyestuffs"; "Disposal of Textile Waste Liquors"; "Textile Strength and Wearing Qualities."

Action of Sodium Hydroxide on Cellulose

"SOME faulty conclusions have arisen concerning the cause and effect of mercerization," says John B. Calkin, fellow of the Textile Foundation, working under the direction of Professor Wilder D. Bancroft at Cornell University, on a study of The Action of Sodium Hydroxide on Cellulose.

"Certain investigators have ascribed to cellulose the property of forming definite chemical compounds with caustic soda, thus accounting for mercerization," states Mr. Calkin's report recently made public. "The first compound claimed by this group, sodium cellulose, is said to occur at about 16–25% caustic solution. Other investigators have arrived at their conclusions on the formation of definite compounds by erroneous application of the phase rule propounded by J. Willard Gibbs.

"Our results and the interpretation of certain other results, indicate that only a partial picture is given by the usual method of attack," Calkin states. "We have found three different curves under the following conditions: (1) native cotton with increasing concentration of caustic solution; (2) native cotton pretreated with sufficiently concentrated caustic solution, washed and air dried; and (3) cotton with decreasing concentration. Cotton pretreated with caustic solution will take up more caustic soda or dye from respective solutions, but there are indications that it is possible, at least in part, to reverse this condition. So far none of our data give evidence of the formation of the sodium cellulose compound.

"The method developed by Vieweg, whereby the caustic taken up is calculated by determining the change in concentration before and after adding the cellulose, has been our line of approach to date. This method gives low values, as it is applicable only where no solvent is taken up by the solid phase. The water from the caustic solution is taken up by the cellulose. Two investigators have published results on centrifuging. We shall try the centrifuging method as a means of ascertaining when all the excess caustic solution has been removed.

"Results of our research show a higher value for the amount taken up from alcoholic caustic solution than from aqueous solution. We have found by the use of cellophane, a non-fibrous form of cellulose, that cellulose takes up little, if any, absolute alcohol when immersed in the pure solvent. With these facts as a basis we are turning our attention to the role of the caustic and of the water in mercerization."

Micro-Analysis of Fibres

"THE forms in which textiles may be produced have been multiplied many times, but as yet the fibre itself is somewhat of a mystery," reports G. Gordon Osborne, Textile Foundation Research Fellow. "Science is now attempting to learn more about the various textile fibres—why and how they exhibit their respective characteristics," continued Mr. Osborne, who is engaged at Massachusetts Institute of Technology, under the direction of Prof. E. R. Schwarz, in a microscopic examination of both the external and internal characteristics of textile fibres.

"The investigation of the physical properties of textile fibres is a field in which little work has been done. What investigation has taken place has been abroad, mainly in Great Britain and Germany, but as yet the surface of the possibilities has scarcely been scratched.

"The work that is taking place at M. I. T. has for its broad purpose the application of scientific research of a fundamental nature to the inherent physical characteristics of textile fibres. The previous work will form the background and much valuable information can be drawn from it and used to advantage. However, the work in the past has centered mainly on obtaining information about the dimensional attributes of the fibres. Thus the information is largely concerning the gross micro-dimensions, in simple, the diameter, cross-sectional area, density, convolutions, length and many others.

"Our work is to center more particularly on the details of external and internal fibre structure. In this respect the study is almost entirely new, what has been done before has been only incompletely pursued. The attempt will be to correlate the microscopically visible or detectable structure with chemical and x-ray analysis and with the results of physical tests as for strength, elasticity, elongation, etc. These other investigations are also being carried out under the auspices of the Textile Foundation.

"Further definite progress in textiles depends upon more specific knowledge of textile raw materials. Upon such knowledge will rest the adoption, improvement, and modification of present manufacturing methods and techniques. For with a more complete understanding of the reactions and workings, that is, the behaviour, of the fibres, their advantages as well as their limitations will be better seen. Efforts now wasted will be conserved and the necessary work to be done will become more efficient."

Abstracts and Bibliography

This section is compiled under the direction of the Committee on Abstracts and Bibliography: Prof. E. R. Schwartz, Chairman, Dr. W. E. Emley, Prof. Louis A. Olney and M. W. Weiss.

Abstracts

I. Fibres (Synthetic and Natural) and Fibre Analysis

Cellulose: Relations Between Refractive Indices and Behavior of Fibres. J. M. Preston. *Trans. Faraday Soc.*, 1933, V. 29, Pt. 1, P. 65–79.

The refractive indices of native, mercerized and regenerated cellulose fibres are related to the orientation of the cellulose micelles of which the fibres are built. Mercerization of cellulose fibres produces changes of the refractive index that depend on whether the fibres are mercerized under tension or without it. The contraction of fibres mercerized without tension can be calculated. The extensibility of regenerated rayons can be calculated from the refractive indices of the fibres. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 1802.) (W)

Cotton: Degree of Ripening of. Oliver Roehrich. *Trans. Faraday Soc.*, 1933, V. 29, P. 218-28; *Chem. Abs.*, 1933, V. 29, P. 2030.

The method of Calvert and Summers for determining the true diameter of a cotton fibre was confirmed. A co-efficient of maturation (percentage of cellulose included in a circle having the same diameter as the fibre) is proposed. For its calculation the average weight per centimeter of Adana and Pima fibres was determined. Mercerized well-matured fibres are easily distinguished from poor ones in a polarizing microscope. (W)

Cotton Hair: Ripening and Dimensions. O. Roehrich. *Trans. Faraday Soc.*, 1933, V. 29, P. 218–227 (*Colloid-Textile Symposium*).

Details are given of the author's methods for determining the measurable characters of cotton and are illustrated by reference to an Adana cotton (Yerli-Koza), the bolls of which do not open on the plant, and a Pima cotton, also from Morocco. Staple diagrams made by the Baer sorter are reproduced and notes are appended on the influence of roller and saw ginning on the staple and on nep formation. Support is given to the method of Calvert and Summers for measuring diameters (D in units of μ). A method is mentioned for determining hair weight per unit length, and

from the data the weight in milligrams per 10 metres (P) is calculated. This is utilized to calculate the area of wall surface, S, from the equation $S = 100P/1.5$, where 1.5 is the density of cellulose and the unit for S is μ^2. From D and S, the total surface S^1 of the hair is calculated, thus $S^1 = \pi D^2/4$, in μ, and finally a coefficient of maturation is obtained from $S \times 100/S^1$. A maturity count, based on mercerisation, is also described, and the account of the tests is completed by reference to tensile tests on single hairs. In the discussion (pp. 227–228) Miss Clegg pointed out the disadvantages of the coefficient of maturation; it would give a general figure but would not reflect those abnormalities of structure which might give rise to neps in spinning. P. Krais showed that if a tube of gelatine hardened by formalin is filled with water, closed at both ends, and then allowed to dry in the air, it convolutes somewhat like a cotton hair. (Copied complete from *J. T. I.*, 1933, May, P. A280.) (S)

DOMESTIC COCOON: BEHAVIOR OF AQUEOUS SOLUTION OF. IX. SWELLING OF SERICIN. X. FREEZING OF SERICIN SOLUTION. H. Kaneko. *J. Agr. Chem. Soc. Japan*, 1932, V. 8, P. 1190–1195, 1196–1200 (Through *Chem Abs.*, 1933, V. 27, P. 1180).

IX—From the sericin solution obtained by boiling cocoons with water, sericin A, but not sericin B, is precipitated by ammonium sulphate at moderate concentration. The swelling of sericin in solutions of the sodium salts of organic acids at 20° has been directly determined. In pure water sericin A swells more than sericin B, possibly owing to regularity of its micelle structure. Sericin B swells considerably in solutions of salts of fatty acids. In acid solution the swelling of sericin is a minimum near the isoelectric point. The swelling in water increases as the temperature rises, especially above 50°.

X—Sericin A and B can be separated by freezing the dilute solution, for sericin A is irreversibly coagulated by freezing, whilst the coagulation of sericin B is reversible. Sericin solution from cocoons having good reeling properties contains a relatively large proportion of sericin A, and exhibits only a small freezing point depression. In solutions of concentration less than 1% the proportion of bound water at pH 6.0 is calculated to be 4–7%. (Copied complete from *J. T. I.*, 1933, May, P. A237.) (S)

FIBRES: FINE STRUCTURE AND MECHANICAL PROPERTIES OF. H. Mark. *Trans. Faraday Soc.*, 1933, V. 29, P. 6–13.

Fibres composed of unoriented rod-shaped micelles show an increased tensile strength upon stretching which is attributed to orientation. The area of micellar contact is thereby increased. The approximate increase in tensile strength can be calculated from the equation $F = F_o(V^{3/1} - p - pV^3)$ where F, F_o are tensile strength of elongated and of original fibre, respectively, p ratio of diameter to length of micelle, V ratio of actual to original length of sample. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 2037.) (W)

FIBRES: SURFACE TENSION AND DEHYDRATION AS CONTRIBUTORY AGENCIES FOR PRODUCTION OF CRIMP. E. Hill. *Trans. Faraday Soc.*, 1933, V. 29, P. 251–4.

Artificial fibres were made by extrusion from simple jets, the fibre material being composed of an irreversible protein gel (egg albumin) which

would behave like wool. The fibre thus obtained resembled wool. The same forces appeared to operate for both. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 2038.) (W)

ISOELECTRIC POINT OF PROTEINS: EFFECT OF ADDITION OF SALTS ON THE. II. S. J. Przylecki and W. Giedroye. *Biochem. J.*, 1931, V. 25, P. 1734–1735.

Evidence is given in support of the opinion that the free charge of protein particles in solution is dependent on the presence of neutral salts and on their concentration; in particular on the valency of their anti- and iso-ions. Experiments on gelatin and serum-albumin are described showing that at concentrations greater than the critical the protein particles may, at the same pH, be supercharged, so that protein solutions are obtained in which, at pH values smaller than that of the isoelectric point, the particles behave as anions, whilst at pH higher than the isoelectric they behave as cations. (Copied complete from *J. T. I.*, Feb., 1933, P. A124.) (S)

ISOELECTRIC POINT OF PROTEINS: EFFECT OF ADDITION OF SALTS ON THE. III. H. Andrzejewski. *Biochem. J.*, 1932, V. 26, P. 1397–1405.

Experiments are described on the influence of electrolytes on the critical concentration, C_k, of alcohol necessary to flocculate gelatin solutions, and on the viscosity of aqueous and aqueous-alcoholic gelatin solutions. The results are discussed as regards the influence of pH and salts on the volume of alcohol necessary, and the influence of electrolytes on viscosity. (Copied complete from *J. T. I.*, Feb., 1933, P. A124.) (S)

KERATIN: CONSTITUTION OF MOLECULE. J. B. Speakman and Mercia C. Hirst. *Trans. Faraday Soc.*, 1933, V. 29, P. 148–72; cf. *Chem. Abs.*, 1932, V. 26, P. 1449, 6147; *T. R. I. Bulletin*, 1932, V. II, No. 6, P. 8.

The micelles of the wool fibre consist of long peptide chains bridged across by salt linkages on the one hand and cystine on the other, the salt linkages being formed from glutamic or aspartic acid and arginine, lysine or histidine. Other types of linkage must exist. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 2038.) (W)

MERCERISED CELLULOSE FIBRES: REFRACTIVE INDEX. J. M. Preston. *Trans. Faraday Soc.*, 1933, V. 29, P. 65–71 (*Colloid-Textile Symposium*).

The view is advanced that the refractive indices of native, mercerised and regenerated cellulose fibres are related quantitatively to the orientation of the micelles. Mercerisation causes changes in the refractive index, the extent of which depend on whether the fibres have been mercerised with or without tension. Values of the higher index, n'_γ, and the lower index, n'_α, are given as follows:

Cotton, native	1.578, 1.532
Cotton, mercerised with tension	1.566, 1.522
Cotton, mercerised without tension	1.554, 1.524
Lilienfeld rayon	1.559, 1.515
Lilienfeld rayon mercerised without tension	1.550, 1.515

The suggestion is made that mounting in media of suitable refractive index might serve as a qualitative test for mercerisation. It is also shown that, from the quantitative effect of tension on the change of refractive indices on mercerisation, the contraction of fibres mercerised without tension can be calculated, with fair agreement with direct observations. The extensibility of rayons can also be calculated from the refractive indices observed on the unloaded filament and at the breaking point. The optical work is criticised and extended in a discussion by W. T. Astbury, van Iterson, R. D. Preston, and E. G. Cox, evidence from X-ray measurements being offered (pp. 71–77). (Copied complete from *J. T. I.*, 1933, May, P. A280.) (S)

REGENERATED CELLULOSE: PROPERTIES. J. Eggert. *Kunststoffe*, 1933, V. 23, P. 25–29.

The nature of cellulose regenerated from viscose is discussed in the light of recent work. Regenerated cellulose approaches more nearly to natural cellulose in its properties after heating and dyeing. The properties may also be changed by compression and, in fact, by applying a pressure of 50 kg. per sq. cm. to a regenerated cellulose, a product is obtained which is less hygroscopic than natural cellulose. The action of the solvents used to dissolve cellulose in the viscose process is discussed with numerous structural formulae. (Copied complete from *J. T. I.*, 1933, May, P. A288.) (S)

SILK: SWELLING OF. W. S. Denham and E. Dickinson. *Trans. Faraday Soc.*, 1933, V. 29, P. 300–305.

The lateral swelling of silk fibroin was determined in solution at pH 1–9, and in air at relative humidities of 0–100. The maximum swelling in air at 100% humidity is 18.7% for Japanese, 16.3% for Italian silk. (Copied complete from *Chem. Abs.*, 1933, V. 27, p. 2038.) (W)

SILK AND WOOL WITH POSITIVE AND NEGATIVE IONS: THE COMBINATION OF. Milton Harris. *Am. Dye. Rptr.*, April 24, 1933, P. 273.

Wool, with an isoelectric point at pH 3.4, combines with negative ferrocyanide ion in solutions ranging in pH up to about 5.0 and with positive nickel ion in solutions ranging in pH down to about 2.0. Similarly silk, with an isoelectric point of pH 2.5, combines with ferrocyanide ion up to about pH 4.0 and with nickel ion down to about pH 1.8. Evidently these amphoteric proteins combine with both positive and negative ions over a certain range on both sides of the isoelectric point. The extent of this 'isoelectric region' depends upon the ions employed as well as upon the protein. The limiting values obtained for the pH at which ions combine with a protein do not necessarily represent the isoelectric point of the protein. (S)

WOOL: ELECTRICAL RESISTANCE OF. M. C. March and Kathleen Earp. *Trans. Faraday Soc.*, 1933, V. 29, P. 173–93.

An apparatus for measuring the specific resistance of wool fibres is described. The influence of time, voltage, relative humidity, percentage moisture content (regain) electrolytes, oil, steaming of stretched and un-

stretched fibres on resistance was determined. The resistance was independent of voltage. The longitudinal conduction in the fibre can be explained by assuming water paths of varying cross sections. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 2038.) (W)

WOOL: SWELLING AND FELTING OF, AS A FUNCTION OF THE pH OF THE WETTING SOLUTION. I. E. Götte and W. Kling. *Kolloid-Z.*, 1933, V. 62, P. 207–13.

An apparatus for measuring linear swelling in the plane of the fibre cross section is described. At 20° and swelling time of 30 minutes, standard wool shows a maximum swelling at pH 3.0, a minimum at 5.8, and very marked swelling at 11.0, due to chemical reaction. At 70°, the maximum is displaced to a very low pH, the minimum to 4.3, and the reaction to 9.0.

II. THE FELTING OF WOOL. (*Ibid.*, P. 213–15.) By means of Arnold's method (cf. *Textile Forschung*, 1930, V. 11, P. 3) the felting of wool was found to decrease linearly with increase in pH. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 2038.) (W)

II. YARNS AND FABRICS

ANALYSIS OF WEIGHTED SILK. R. T. Mease. *Bur. Stds. J. Rsch.*, Research Paper No. 498, 1932, V. 9, P. 669; *Tex. Rsch.*, 1933, V. 3, No. 4, P. 194–206.

In the analysis of silk the most important constituent to evaluate is the silk fibroin. A strip of the full width of the fabric (3 to 5 grms.) is dried by spreading it loosely on a watch-glass in an electric oven at 105° to 110° C. for 1½ hours, and weighed in a tared weighing bottle. The dry weight is called weight A. Soluble finishing materials are removed from the dry material by two immersions of two minutes each in 30-c.c. portions of ether, at laboratory temperature, with squeezing after each immersion. The fabric is then similarly treated with ethyl alcohol at 50° to 60° C., followed by 20 minutes' immersion in water at 65° to 70° C., and three half-minute immersions, after which it is well squeezed by hand and dried as before. The weight at this stage is weight B; $\frac{(A - B) \times 100}{A}$ = the percentage of soluble finishing materials.

The more firmly held weighting and finishing materials are now removed by immersing the treated fabric for 20 minutes in 90 to 100 times its weight of a solution containing 2 per cent. hydrofluoric and 2 per cent. hydrochloric acid—equivalent to N in HF and N/2 in HCl at 55° C. ± 1° C. (This solution should be contained in a Pyrex vessel coated with ozokerite.) The acid solution is poured off, and the material is rinsed with two portions of water at 55° to 60° C., followed by handsqueezing after each rinsing. This acid treatment is repeated. The squeezed fabric is then given a similar treatment in which the acid is replaced by 2 per cent. sodium carbonate solution, followed by two more acid treatments and several rinsings with water, and is then dried as before (weight C). The percentage of silk fibroin equals $\frac{C \times 100}{A}$.

The various immersions are carried out in a circular oil-bath, capable of holding several beakers, so that several analyses can be carried out simul-

taneously. Qualitative tests are briefly described for silica, lead, aluminium, tin, zinc and phosphage, and references are given in case further details should be required. Inorganic impurities can be detected on burning a small piece of the fabric. Any ash will retain the form of the original threads. (Copied complete from *The Analyst*, March, 1933, P. 175.) (S)

MILDEW ON RAYON FABRICS: OCCURRENCE OF. P. Dory. *L'Ind. Text.*, 1933, V. 50, P. 102.

Mildew stains on rayon fabrics are attributed chiefly to the presence of residues of gelatin size which serve as food for micro-organisms. The defect is observed most frequently on fabrics with high weft densities or prepared from yarns of high twist; the stains are generally found in folds or in the seams of garments. Infection may come from the liquids used in processing or from dust particles. Thorough desizing is necessary for the avoidance of this defect. The use of linseed oil sizes in place of gelatin has been recommended. (Copied complete from *J. T. I.*, 1933, May, P. A283.) (S)

YARNS: THE EFFECT OF REGAIN UPON THE PROPERTIES OF WOOLEN. Herbert J. Ball. *L. T. I. Bull.*, Series 36, No. 4, May, 1933.

Purpose of the test was to ascertain the effect of regain upon the breaking strength and elongation of woolen yarns ranging in number from .74 to 4.98. The data is determined in terms of strength factor; breaking strength per skein multiplied by the run number. From the plotted data using the strengths at 16% regain as standard the rate of chance of strength factor per percent regain is equal to — 0.5 (Run number) — 2.25. As a generalization it may be therefore stated that as woolen yarns become finer there is a proportionate increase in their rate of decrease in strength with increasing regain. It was also found that the elongation expressed as percent increased with moisture as a straight line function. (S)

III. CHEMICAL AND OTHER PROCESSING (NOT OTHERWISE CLASSIFIED)

DRY-CLEANING SOLUTIONS: DETERMINATION OF SOAP AND FATTY ACIDS IN. E. E. Hughes. *Natl. Cleaner & Dyer*, 1933, V. 24, No. 2, P. 39–40.

An analysis of three typical dry-cleaning soaps of known composition shows that the amount of soap and fatty acid present in dry-cleaning solutions can be determined by titrating to neutrality with approximately tenth normal alcoholic potass. hydroxide with thymol blue as an indicator; calculating the free oleic acid corresponding to the alkali used; titrating the solution further with fifth normal hydrochloric acid after the addition of bromthymol blue indicator; calculating the potassium oleate corresponding to the acid, and subtracting this amount from the potass. oleate equivalent of the oleic acid found. The difference represents the soap originally present. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 2039.) (W)

DYEING OF COTTON. J. Rehner, Jr. *Rev. soc. brasil. chim.*, 1931, V. 2, P. 503–6; cf. *Am. Dye. Rptr.*, 1930, V. 19, P. 465.

The fixing of dyes on cellulose or protein is considered to be a chemical reaction, since, when basic or acid dyes are employed the pH of the solution

falls or decreases, respectively, combination between the dye and the protein, etc. residue is indicated. Bitancourt's suggestion (*Chem. Abs.*, V. 25, P. 4407) that the dye combines with the hydrogen ion from the cellulose, cannot be general in application. (Copied complete from Chem. Abs., 1933, V. 27, P. 2037.) (W)

SULFONATED OILS: DETERMINATION OF SULFATE IONS IN. Fritz Neuber and Hans Wächter. *Z. anal. Chem.*, 1933, V. 91, P. 425–32.

Dissolve about 10 g. of the oil in 100 cc. of alcohol and heat just below the boiling temperature until the resulting Na_2SO_4 precipitate has settled well. With dark oils add at the start 1 cc. of glacial acetic acid. Filter and wash the alkali sulfate precipitate with hot alcohol. Dissolve in hot water and carry out the usual gravimetric determination as $BaSO_4$. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 1842.) (W)

IV. RESEARCH METHODS AND APPARATUS

APPLICATION OF INFRA-RED PHOTOGRAPHY TO TEXTILES. P. W. Cunliffe. *J. Soc. Dyers and Col.*, Mar., 1933, P. 73.

The development of the new infra-red sensitive photographic plates has made possible the application of infra-red photography to textiles inexpensively. Of the possible uses one is the detection of faults in dark colored materials. Another is the selection of dyestuffs for use with dark cloths which are to be as cool as possible in the summer. This latter is accomplished by determining the amount of infra-red or light rays reflected. (S)

DIASTASE: VISCOSIMETRIC MEASUREMENT OF STARCH LIQUEFYING POWER. J. J. Willaman, E. W. Clark and O. B. Hager. *Biochem. Z.*, 1933, V. 258, P. 94–101.

Changes are introduced into the viscosimetric method of estimating the liquefying power of diastase preparations for starch. These involve the shape of the viscosity pipette, the nature of the buffer solution, and the use of formaldehyde to prevent enzyme action. Liquefying power is defined as the weight of starch in grams liquefied by 1 gm. of enzyme preparation in 1 hr. at 40° C. and a formula is given which connects this with the number of milligrams of the preparation taken in the test and the percentage fall in viscosity. The following numbers are recorded for commercial desizing agents, etc.: Takadiastase 12,180; Polyzime P. 86,400; Rapidase 1330; Diastafor 2160; Degomma S (tryptic pancreatin) 41,900; and Degomma DL (diastatic pancreatin) 205,000. (Copied complete from *J. T. I.*, 1933, May, P. A285.) (S)

FLUORESCENCE MICROSCOPY APPARATUS. C. Reichart (Vienna). *J. Sci. Instr.*, 1933, V. 10, P. 86–88.

An apparatus is described for fluorescence microscopy, in which the source of light emits rays forming an almost continuous band of lines in the range 2200–4400 A. The actual light source consists of an arc lamp equipped with special iron electrodes. The visible portion of the light is shut off by a two-stage filter (smoked glass and copper sulphate solution). The apparatus can be used on any existing microscope, it being merely

necessary to change the mirror and the condenser and provide a suitable excluding filter. (Copied complete from *J. T. I.*, 1933, May, P. A281.) (S)

HEXOSES: COLOUR TESTS. J. H. Foulger. *J. Biol. Chem.*, 1933, V. 99, P. 207–211.

Two new reagents, containing respectively urea and guanidine dissolved in a stannous chloride-sulphuric acid solution, are found to give definite colour reactions with the simple hexoses. The reagent containing urea differentiates between keto-hexoses and aldo-hexoses. With the former it gives on boiling a greenish-blue solution, changing to bluish-violet on cooling, whilst aldo-hexoses give yellow or olive-green solutions changing to reddish-blue on cooling. Pentoses give intense yellow colours. The reagent containing guanidine gives a distinct colour for each sugar without a definite distinction between keto-hexoses and aldo-hexoses. Absorption spectra are shown. (Copied complete from *J. T. I.*, 1933, May, P. A290.) (S)

LIGHTING APPARATUS: SURFACE MICROSCOPY. H. Wagner and M. Zipfel. *Chem. Fabr.*, 1932, 421–423.

Three kinds of lighting apparatus are described, the illuminator with light falling on the object at an angle, that with the light falling vertically, and the dark field illuminator. Their particular applications to different objects are discussed, and photographs are given of graphite and blanc fixe illuminated by all three methods. (Copied complete from *J. T. I.*, Feb., 1933, P. A118.) (S)

Bibliography

I. FIBRES (SYNTHETIC AND NATURAL) AND FIBRE ANALYSIS

CELLULOSE: MODERN IDEAS ON THE STRUCTURE OF. John H. Skinkle. *Am. Dye. Rptr.*, April 24, 1933, P. 267.

A very brief review of certain of the literature dealing with cellulose structure but omitting all references. (S)

CELLULOSE: MODIFICATION OF NATURAL, BY SWELLING AND DEGRADATION. S. M. Neale. *Trans. Faraday Soc.*, 1933, V. 29, P. 228–38.

The purpose of this review is to show that cotton purified by a dilute alkali boil is a material of more or less definite properties, and to indicate the essential lines along which this material can be modified. A compilation of constants is included. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 2031.) (W)

VISCOSE RAYON: NOTE ON THE DETERMINATION OF, IN MIXTURES OF ARTIFICIAL SILK AND COTTON WASTE. P. McGregor and C. F. K. Fryd. *J. T. I.*, Feb., 1933, P. T103.

Use of a commercial solution of calcium thiocyanate containing about 80 g. per 100 ml. Exact detail of use is given. (S)

II. Yarns and Fabrics

Cotton to Yarn Quality and Character: The Relation of. Dr. A. J. Turner. *Tex. Mfr.*, Feb., 1933, P. 47.

Yarn qualities have a relation to cotton fibre properties, as may be seen broadly from particulars of fibres and yarns made, but exact relationship has not yet been determined. Irregularity depending on the 'drafting wave' is a factor limiting fineness of spinning, and it prevents prediction of spinning and yarn qualities from fibre properties. (S)

Dyed Wool: Fastness to Washing. Bertil A. Ryberg. *Am. Dye. Rptr.*, April 24, 1933, P. 275.

A brief statement of the selection by a sub-committee of the A. A. T. C. C. on fastness tests for dyeing wool. The dyes selected for four classes including the dyeing method for each are given. (S)

Latex: The Impregnation of Textiles with. W. J. R. Hauser. *Rubber Age*, Jan. 10, 1933, P. 242.

A brief discussion illustrated with a number of photomicrographs illustrating the rubber into the textile. (S)

Rubber Thread. E. A. Murphy. *India Rubber World*, April, 1933, P. 25.

A comparison of the extruded with the cut filament. (S)

Yarns Without Twist. Anon. *Tex. Mfr.*, March, 1933, P. 90.

A brief statement regarding British patent 361,037. The cotton fibres are held in parallel position with an adhesive. (S)

III. Chemical and Other Processing (Not Otherwise Classified)

Aspergillus Niger: Influence of Organic Substances on Growth. L. E. Kiessling and A. Schmidt. *Chem. Zentr.*, 1932, V. ii, P. 3008 (from *Wiss. Arch. Landwirtschaft. Abt. A. Pflanzenbau*, 1932, V. 9, P. 293–305).

Additions of filter paper, parchment, peat, pine chips, lignin, sodium silicate, barley roots, and sugar beet leaves produce increases in the mycelial weight of Aspergillus niger on nutrient solutions and on soils. (Copied from *J. T. I.*, Feb., 1933, P. A120.) (S)

Dye Particle: Measurement. J. Gicklhorn. *Mikrochem.*, 1932, V. 11, P. 369–428.

The application of physical and chemical microtests in biology and medicine is reviewed, methods for determining density, viscosity, conductivity, and so forth are discussed, and particular attention is given to the study of particle size in solutions of dyes and colourless substances. (Copied complete from *J. T. I.*, Feb., 1933, P. A118.) (S)

DYEING: THEORY OF. Influence of Acid Dyes on Animal Fibres. E. Elöd. *Trans. Faraday Soc.*, 1933, V. 29, P. 327–47.

A review, mostly of the studies of the author and collaborators. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 2037.) (W)

DYESTUFFS: RAISING THE SOLUBILITY OF, BY NEUTRAL SALTS. W. Ostwald. *Trans. Faraday Soc.*, 1933, V. 29, P. 347–55; *Chem. Abs.*, 1933, V. 27, P. 2038.

Review and general discussion. (W)

DYESTUFF SOLUTIONS: PHYSICAL CHEMISTRY OF. S. M. Neale. *Am. Dye. Rptr.*, April 10, 1933, P. 237.

A brief statement of the present state of knowledge of the state of dyestuffs in solution. Dyestuffs apparently must be treated as colloidal electrolytes. (S)

FIBROUS MATERIALS: MECHANICAL EQUIPMENT FOR CONTINUOUS FERMENTATION OF. A. M. Buswell and C. S. Boruff. *J. Ind. and Eng. Chem.*, Feb., 1933, P. 147.

An apparatus has been designed which overcomes the operating difficulties in continuously fermenting fibrous materials, such as cornstalks, sewage screenings, paunch manure, and the like. Gas (carbon dioxide and methane) recovery data and operating technic are given. This type of tank should be applicable to the alcoholic, acid, or acetonic fermentation of fibrous materials. (S).

IV. RESEARCH METHODS AND APPARATUS

COLORS: A SYSTEM OF STANDARDIZING. D. P. Dieterle. *Am. Dye. Rptr.*, April 10, 1933, P. 253.

An application of a mathematical analysis to the usual color triangle. (S)

HYDROGEN ION MEASUREMENT AND CONTROL. P. L. Mann. *Am. Dye. Rptr.*, Feb. 27, 1933, P. 167.

A brief review of the importance of hydrogen ion determination and control. Nothing new is stated. (S)

TEXTILE TESTING: SOME ELEMENTARY FACTS IN. R. H. Adams. *Am. Dye. Rptr.*, March 27, 1933, P. 212.

A somewhat general discussion of the usefulness of a testing laboratory in the mill with particulars of the Callaway Mills laboratory installations. (S)

V. ECONOMICS AND MISCELLANY

RESEARCH: SCIENTIFIC AND INDUSTRIAL. *Tex. Mfr.*, Mar., 1933, P. 93.

A report of work done by the cotton, wool, linen and silk industries research associations noted in a brief abstract of the 193 page pamphlet the outstanding achievements. (S)